

Materials Scale-up and Cell Performance Analysis

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LBL

DOE AMR

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Project ID #
ES029

Timeline

- Start: June 2009
- End: September 2012
- Percent complete: 30%

Budget

- Total project funding: \$580 k
- Funding received in FY09: \$290 k
- Funding for FY10: \$290 k

Supports $1\frac{1}{8}$ Research Associates

Barriers

- Barriers addressed
 - High Cost (<150 \$/kWh)
 - Low Energy Density (>230 Wh/l)

Partners

- BATT PIs
 - Dillon (NREL)
 - Doeff (LBNL)
 - Dudney (ORNL)
 - Ceder (MIT)
 - Kumta (Pitt. U.)
 - Lucht (U.R.I.)
 - Thackeray (ANL)
 - Zaghib (HQ)

Objective

Evaluate New Materials Being Developed in the BATT Program Against DOE Goals and Baseline Performance Markers

- For FY '10 we expected to evaluate at least four new materials.
- Such evaluation provides guidance to the researchers as to what degree they have surpassed the baseline performance and how much farther we need to go toward meeting the DOE/USABC Performance Targets.
- Tracking progress is critically important to making progress.

Approach

1. Make inquiry to BATT PIs for new materials they deem ready for the next step
 - Scale-up to 10 g batch
 - Evaluation in full cell
2. Test in half cell against Li using electrode fabrication techniques developed in BATT program.
 1. If capacity density and first cycle irreversible capacity improve on baseline then go to next step.
 2. Measure rate capability at different C-rates at a reasonable loading. If improvements over baseline, then go to next step.
 3. Work with BATT cell modelers to design electrodes for full cells.
3. Evaluate cycle life in full cells.
 - Attempt to identify performance attributes and limitations.
4. Report performance results
 1. To PI
 2. At semi-annual ABRT meetings.
 3. If performance is favorable, at DOE AMR meeting.

**Received 8 responses from BATT PIs
expressing interest in scale-up and
evaluation**

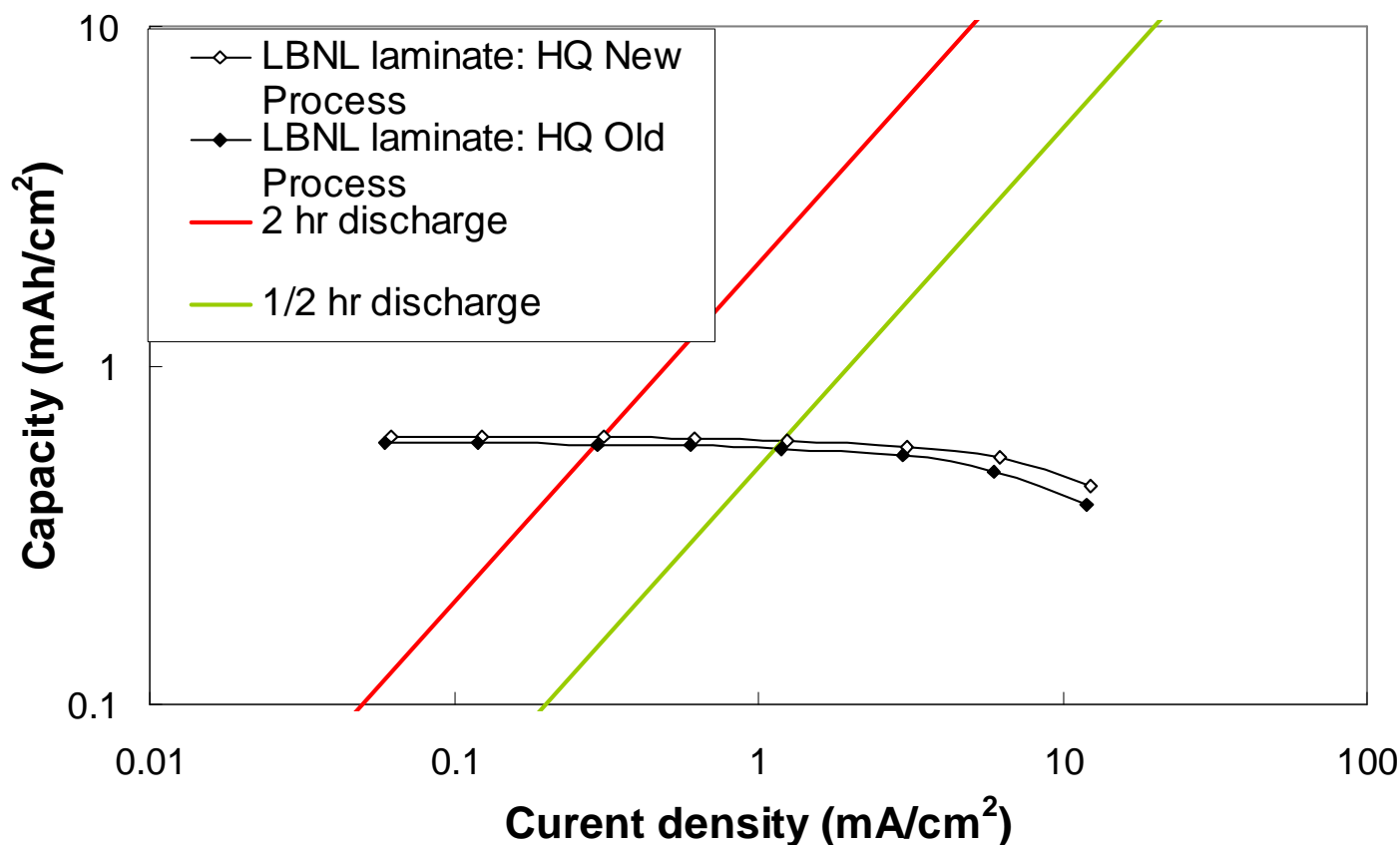
- Completed evaluation of 4 materials.
- In the process of testing 3 materials
- Will work with the last PI in the coming months.

Technical Accomplishments

Investigator	Institution	Material	Barrier	Feedback	Status
Respondents in FY09					
G. Ceder	Massachusetts Inst. of Technology	High-rate LiFePO_4	High system cost	We made the material w/ their guidance	Cycle-life testing
M. Thackeray	Argonne National Laboratory	High-capacity NCM material	Low energy density	Sent us materials and electrode formulations	Cycle-life testing
N. Dudney	Oakridge National Laboratory	LiFePO_4 in carbon mat – no Cu cur. col.	High system cost	Sent us anodes	Tests complete (low cap. dens.)
M. Doeff	Lawrence Berkeley Nat. Laboratory	Al-doped NCM material	High material cost	We will make material w/ their guidance	To be initiated
P. Kumta	University of Pittsburgh	Si-C nanocomposite	Low energy density	Sent 1 st gen anodes.	Tests complete (high 1st cycle ICL)
K. Zaghib	Hydro-Québec	Lower cost LiFePO_4	High cost	Sent 50 g of powder and laminates	Cycle-life testing
A. Dillon	National Renewable Energy Lab.	High capacity MoO_3 anode	Higher energy density	Sent us anodes	Tests complete (high 1st cycle ICL)
B. Lucht	University of Rhode Island	$\text{LiPF}_4\text{C}_2\text{O}_4$ thermally stable salt	Poor high temperature performance	Sent us 10 g of salt	Tests complete (high 1st cycle ICL)

Technical Accomplishments

A comparison of two HQ materials: one received a year ago and one received 6 months ago. The new material was made *via* a less costly process.



When making comparisons, one needs to fabricate electrodes of identical loadings.

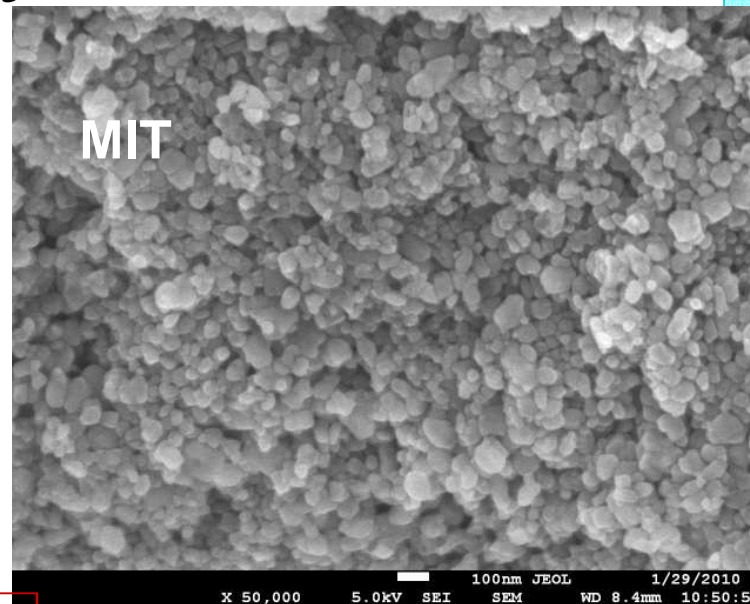
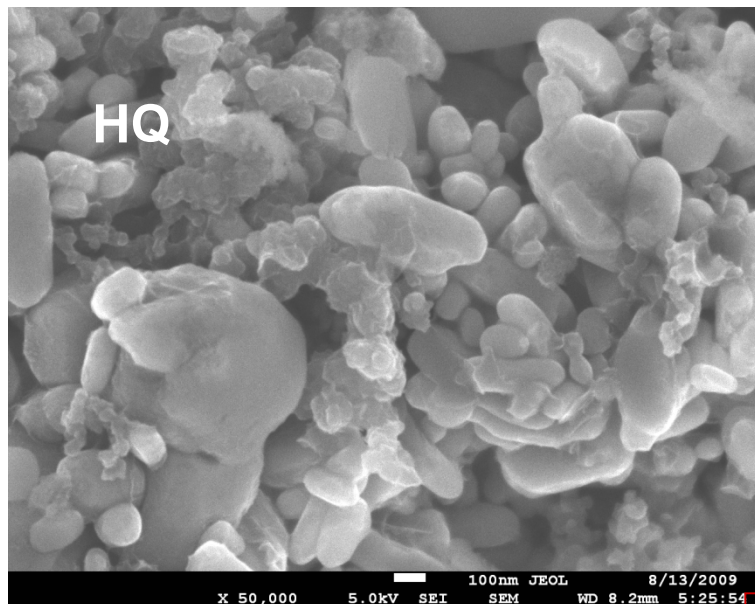
MIT Nano-LiFePO₄ Material

- We sent two scientists and Gao Liu to MIT for one week at the end of November during the MRS meeting in Boston
 - We made a batch of material there.
 - We were given ca. 1 gram of powder.
 - We were given an electrode laminate and Swagelok hardware for cell testing.
- Since then we have:
 - Made several 7 g batches of the material.
 - Characterized the materials with SEM, BET, and PSA.
 - Made laminates following their recipe using PTFE
 - Made laminates following our recipe using PVdF.
 - Exchanged testing results.
 - Prof. Ceder visited our lab in March.
 - Sent samples out for XPS analysis.
 - Been working to make higher rate electrodes.
 - Been working with the Molecular Foundry to do further surface analysis.

This has been a very open collaboration between all scientists involved!

SEM - Primary Particles

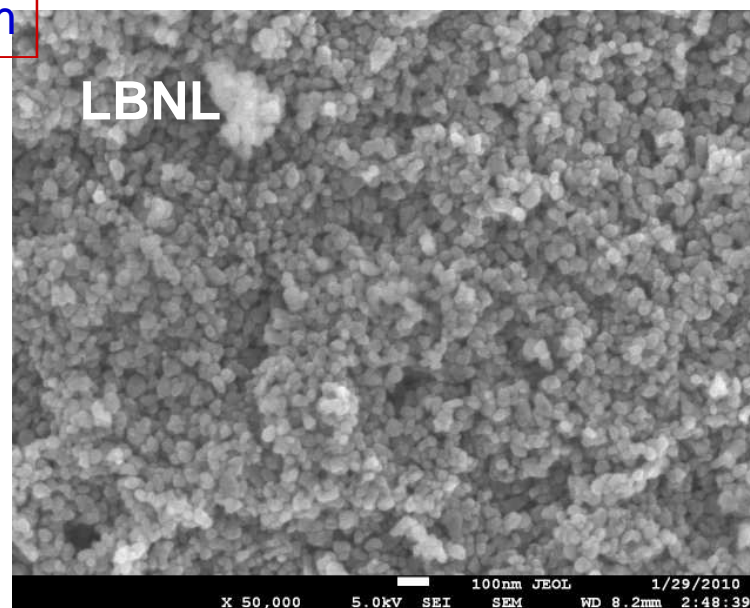
X. Song



— 100 nm

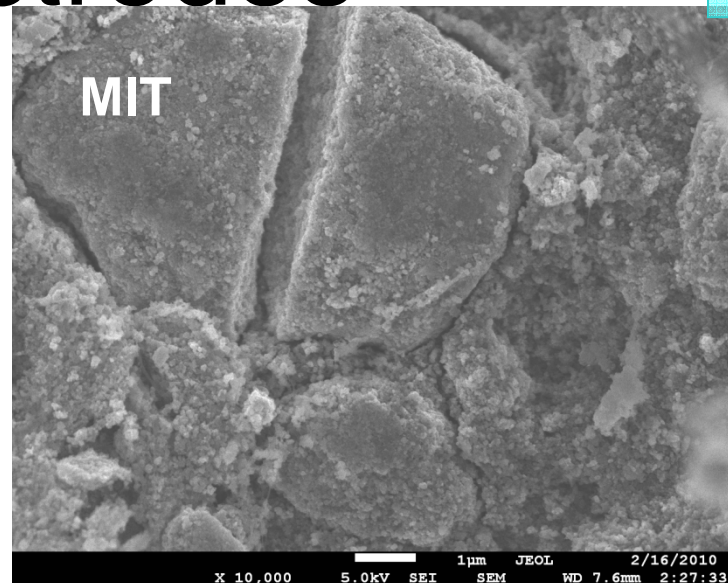
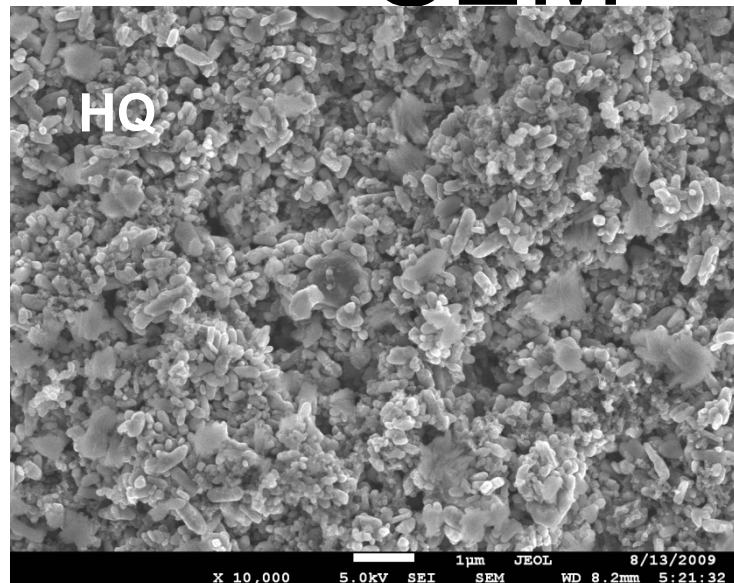
SEMs at 100 nm

- For the HQ material, primary particles range from 50 to 500 nm
- For the MIT and LBNL, material primary particles are around ca. 25 nm



SEM - Electrodes

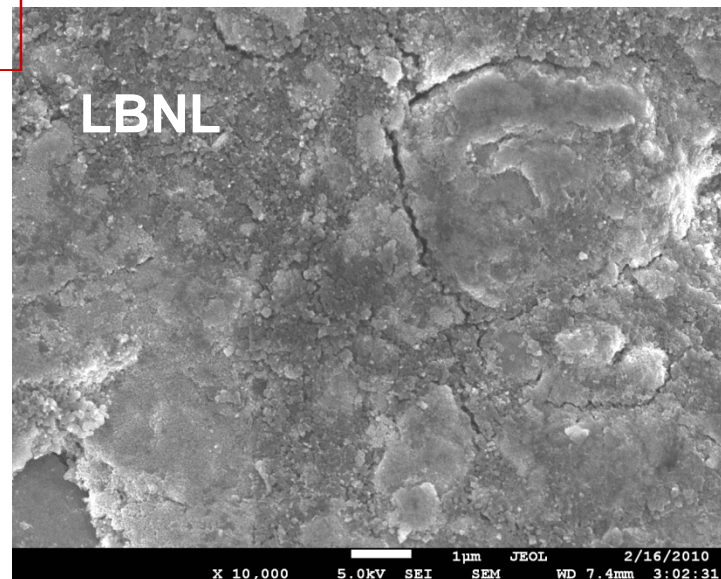
X. Song



— 1 μm

Electrodes

- HQ shows uniform mixing of additives and primary particles.
- For the MIT and LBNL, a fraction of the powder was still clumped together.



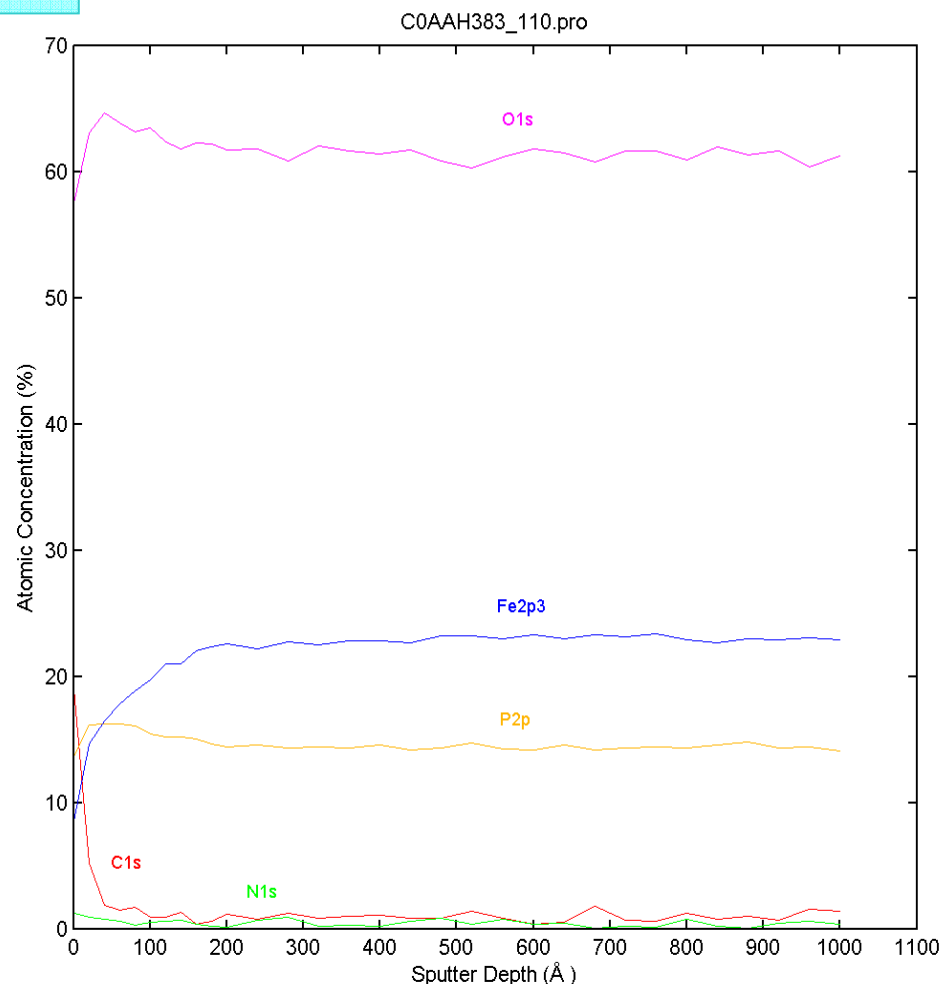


The MIT nano-LiFePO₄ is jet black.

- EDX indicated that there is some carbon on the surface.
- We sent this material out for XPS analysis, along with a sample of HQ's material.

XPS & Ion Sputtering of MIT LiFePO_4

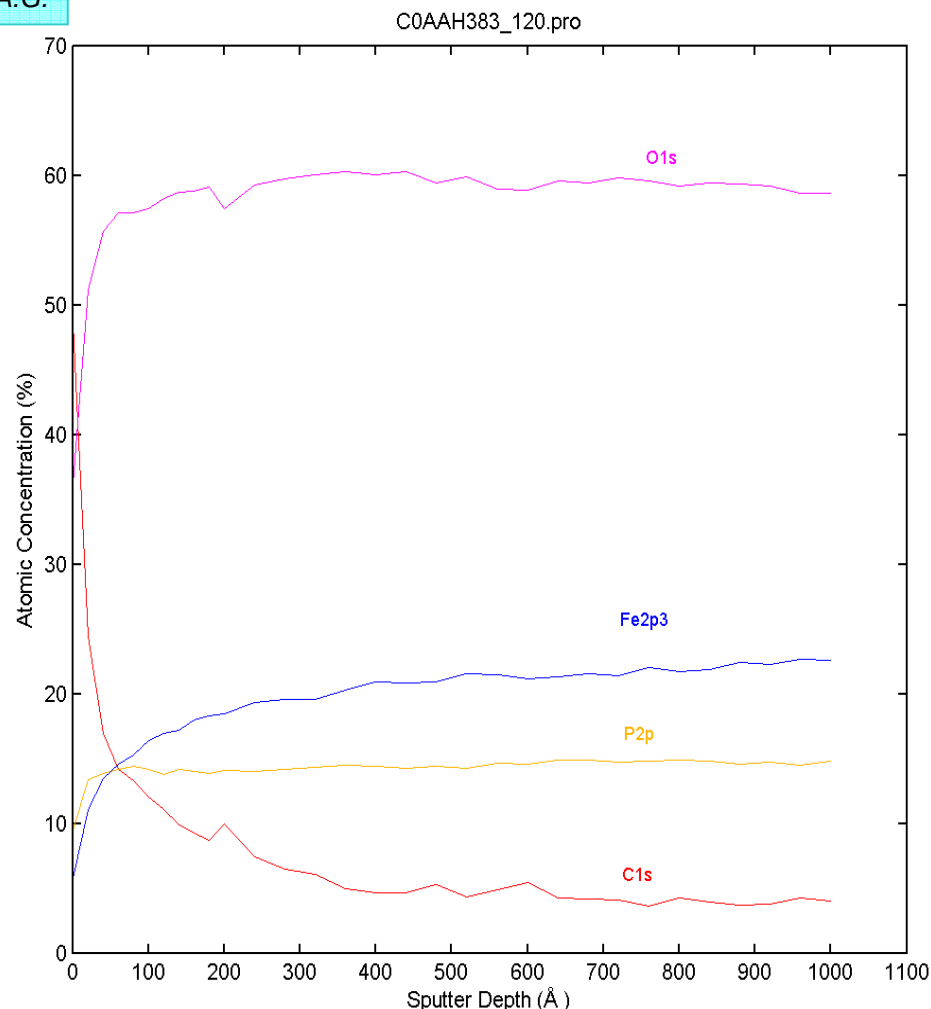
E.A.G.



- Points of interest
 - Slight **carbon** coating of **1 to 2 nm**, believed to be residue of $\text{FeC}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$ precursor.
 - Oxygen and phosphorous are uniform and of the same ratio from surface to interior **P:O = 1:4**.
 - Accumulation of data suggests this sample is coated with **2 to 5 nm** of **Li_3PO_4** .

XPS & Ion Sputtering of HQ LiFePO₄

E.A.G.



- Points of interest
 - Carbon coating 5 nm thick, believed to be an elemental carbon.
 - Oxygen and phosphorous are uniform and of the same ratio from surface to interior $P:O = 1:4$.
 - There may be a thin coating of Li₃PO₄ on the surface of this material as well, underneath the carbon.

Collaborations

Investigator	Institution	Interaction	Investigator	Institution	Interaction
G. Ceder	MIT	We make material w/ their guidance	P. Kumta	U.Pitt.	Will send 30 to 100 g of 1 st gen anodes
M. Thackeray	ANL	Will send us materials and electrode formulations	K. Zaghib	H.Q.	Will send 30 to 100 g of powder and laminates.
N. Dudney	ORNL	Will send electrodes	A. Dillon	NREL	Will send laminates
M. Doeff	LBL	We make material w/ their guidance	B. Lucht	U.R.I	Will send 10 g of salt

We are also working with [V. Srinivasan](#) of the Modeling Group of the BATT program to design cells.

[P. Ross](#) helped us with acquiring and interpreting the XPS data.

Acknowledgment

The majority of this work was carried out by [Jin Chong](#) and [Honghe Zheng](#).

Proposed Future Work

- Rest of this year
 - MIT material looks promising as a high rate material
 - Finish electrode processing and testing.
 - Work with BATT Modeling Group and MIT to determine best automotive application.
 - Design electrodes and test cells to that application.
 - We have additional experiments planned to understand why this sample is black.
 - ANL's materials are in early phases of evaluation
 - Finish initial tests.
 - Decision point - If improvement over baseline materials, we will:
 - Work with BATT Modeling Group and ANL to determine best automotive application.
 - Design electrodes and test cells to that application.
 - H.Q. laminates are in the early stages of evaluation
 - Finish initial tests.
 - Decision point - If improvement over baseline materials, we will:
 - Work with BATT Modeling Group and ANL to determine best automotive application.
 - Design electrodes and test cells to that application.
 - Low-Co NCM is important, scale-up LBNL's Al-substituted NCM.
- All milestones completed

Summary

- Eight BATT PIs answered the call for materials evaluation.
 - 4 sent laminates
 - 1 sent powders
 - 1 sent both
 - 2 asked us for assistance in scale-up
- Benefits and limitations of materials have been confirmed and conveyed back to PIs.
- New, low-cost HQ material performs as well as previous material.
- MIT material needs further processing to make good electrodes – this work has begun.
- Both MIT and HQ materials have coatings and perform well.
 - HQ's has 100 nm primary particles with a 5 nm carbon coating that improves the electronic conductivity and leads to good electrode performance.
 - MIT's has 30 nm primary particles with a 5 nm phosphate coating that mitigates secondary particle formation and minimizes solid-state diffusion limitations that leads to good electrode performance.

Supplemental Slides

Responses to Previous Year Reviewers' Comments

LBNL

New project; started June 2009.

Publications and Presentations

LBNL

None to date.

- We will be able to make electrodes with the MIT material using acetylene black as the conductive additive
 - Will require some effort in determining best mixing strategies.
 - Preliminary results suggest that this will not be a problem.